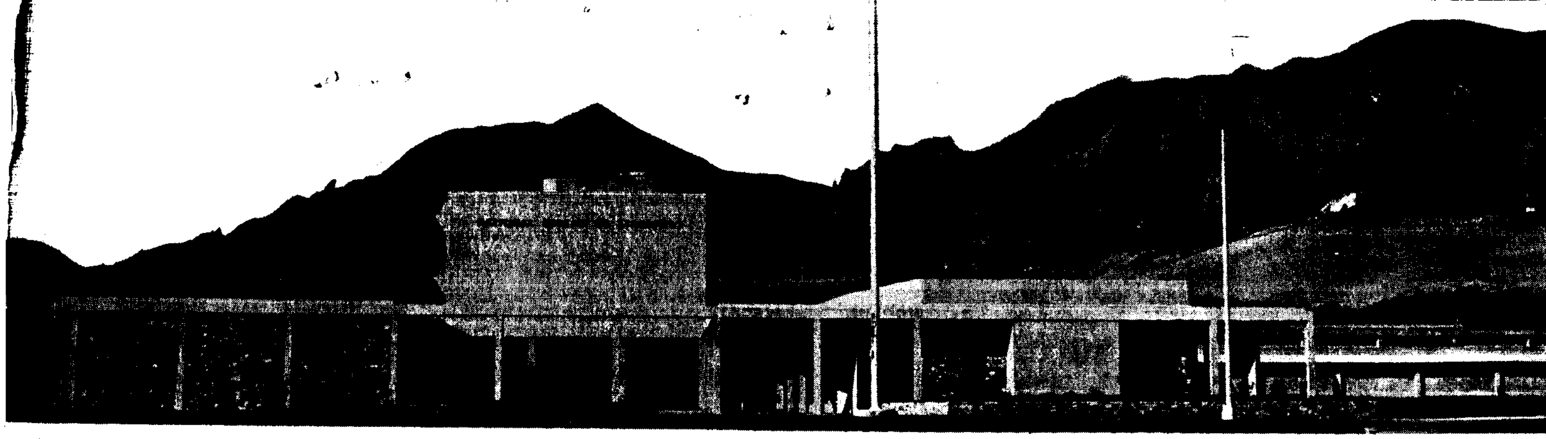

NBS REPORT

9268

R-45

# TWENTY-FOURTH PROGRESS REPORT

to

National Aeronautics and Space Administration

on

Cryogenic Research and Development

for

Period Ending December 31, 1966

NBS




U. S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

BOULDER LABORATORIES

Boulder, Colorado

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# NATIONAL BUREAU OF STANDARDS REPORT

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TWENTY-FOURTH PROGRESS REPORT

to

National Aeronautics and Space Administration

on

Cryogenic Research and Development

for

Period Ending December 31, 1966

Cryogenics Division
Institute for Materials Research
National Bureau of Standards
Boulder, Colorado



U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

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# 1. Physical Properties of Cryogenic Fluids

## 1.0 General Comments

Personnel contributing during this period were D. E. Diller, W. J. Hall, L. A. Weber, and B. A. Younglove.

## 1.1 Parahydrogen

### 1.1.1 Dielectric Measurements on Solid Parahydrogen and Melting Pressures of Parahydrogen

Preliminary data on the dielectric constant for solid parahydrogen, consisting of eight measurements between 15.0°K and 16.4°K, show good internal precision. They average about 0.8% below calculated values based on the Clausius-Mossotti equation and extrapolated values of the dielectric constant and polarizability from J. W. Stewart's work on liquid parahydrogen. Further measurements will test for reproducibility of these measurements and will be extended to other temperatures.

About 25 pressures on the melting curve of parahydrogen have been measured and compared to values calculated from R. D. Goodwin's equation. Excellent agreement is obtained in the temperature range 15.8°K to 17.8°K, the latter temperature being the highest taken in this test. Below 15.8°K the measured pressures fall below those calculated, this difference becoming larger as the triple point temperature is approached and amounting to more than 1.5% in pressure. These data will be fitted to the Simon equation using the triple point temperature as a parameter. Additional data on the melting curve will be taken, especially at temperatures above 17.8°K and below 15.8°K.

### 1.1.2 Refractive Index Measurements on Gaseous Hydrogen

Refractive index measurements on normal hydrogen at 298°K and low densities ($\rho \leq 0.01$ g/cm$^3$) have been completed. Measurements on normal hydrogen at 98°K and on parahydrogen at 100°K and at somewhat higher densities ($\rho \leq 0.04$ g/cm$^3$) are now being made. From comparisons of the temperature, density and composition dependence of the Lorentz-Lorenz function, the following tentative inferences may be drawn:

1) The polarizability of normal hydrogen is about 0.1% smaller at 98°K than at 298°K.

2) The density dependence of L-L for normal and parahydrogen is very similar at 98°K and low densities. Both show an initial increase with density of about 5 parts in 10,000.

   Densities of normal hydrogen at 98°K measured by Michels et al. differ from those compiled by Wooley, Scott, and Brickwedde by up to 0.4%. Based on a comparison of the measurements of L-L for normal and parahydrogen and on expectations from theory, the latter (WSB) appear to be much more correct.

3) The density of normal hydrogen at 98°K is about 0.15% smaller than that of parahydrogen. This inference is based on the measured difference in L-L (0.3%) and on the polarizability difference (0.15%) obtained from the literature.

We plan to make measurements on parahydrogen next at 35°K and densities up to 0.08 g/cm$^3$. This will double the density range obtained so far.

### 1.1.3 TAB Code

The final report on the TAB codes is in preparation. It will be issued as a special report as soon as possible.

## 1.2 Oxygen

### 1.2.1 P-V-T Measurements on Oxygen

Data-taking has been completed, with the possible exception of a few supplemental runs to determine behavior in certain specific regions, such as the region close to saturation, or the critical region. We now have approximately 1500 P-V-T data points in the region 56°-300°K and pressures to 330 atm.

Very precise measurements of the melting curve were made at pressures up to 160 atm and were found to be in excellent agreement with the less precise data of Mills and Grilly [1], which extend to 3500 atm. Application of the Simon melting equation allowed an independent determination of the triple point temperature, which was found to be $54.350_8$°K. The vapor pressure measurements are in very good agreement with the curve given by R. B. Stewart [2]. Present efforts involve fitting all the data with analytic curves for the purpose of smoothing and interpolation.

No further reports on properties of oxygen will appear in this series, since support from the present contract ceased on October 31, 1966. This work will be carried on under NASA-OART Contract No. R-06-006-046, NBS Project 3150445, and will be reported elsewhere. Future work will include calorimetric measurements of specific heats and preparation of detailed tables and charts of derived thermofunctions.

## 1.3 Thermodynamic Property Charts of Saturated Liquid Parahydrogen

In response to a specific request by the sponsor, a set of six multi-colored 20" x 30" temperature-entropy diagrams was developed

---

[1] Mills, R. L., and E. R. Grilly, Physical Review 99, 2, 400 (1955).

[2] Stewart, R. B., Thesis, The Thermodynamic Properties of Oxygen Dept. of Mechanical Engineering, U. of Iowa, (June 1966).

covering the temperature range of 29.2°R to 42.5°R with pressures to 100 psia and mixtures of the liquid and vapor phases to 0.003 quality (roughly 25 volume percent vapor). These charts should be useful to the pump designer, for flowmeter analysis, and in bubble chamber work. To achieve greatest utility, the engineering (British) system of units was used. The range and relative location of these diagrams are shown in figure 1.

The charts and accompanying report may be obtained from the Cryogenic Data Center - identified as "Thermodynamic Property Charts of Saturated Liquid Parahydrogen in British Units" by R. D. McCarty and H. M. Roder - NBS Report 9263 (November 1966).

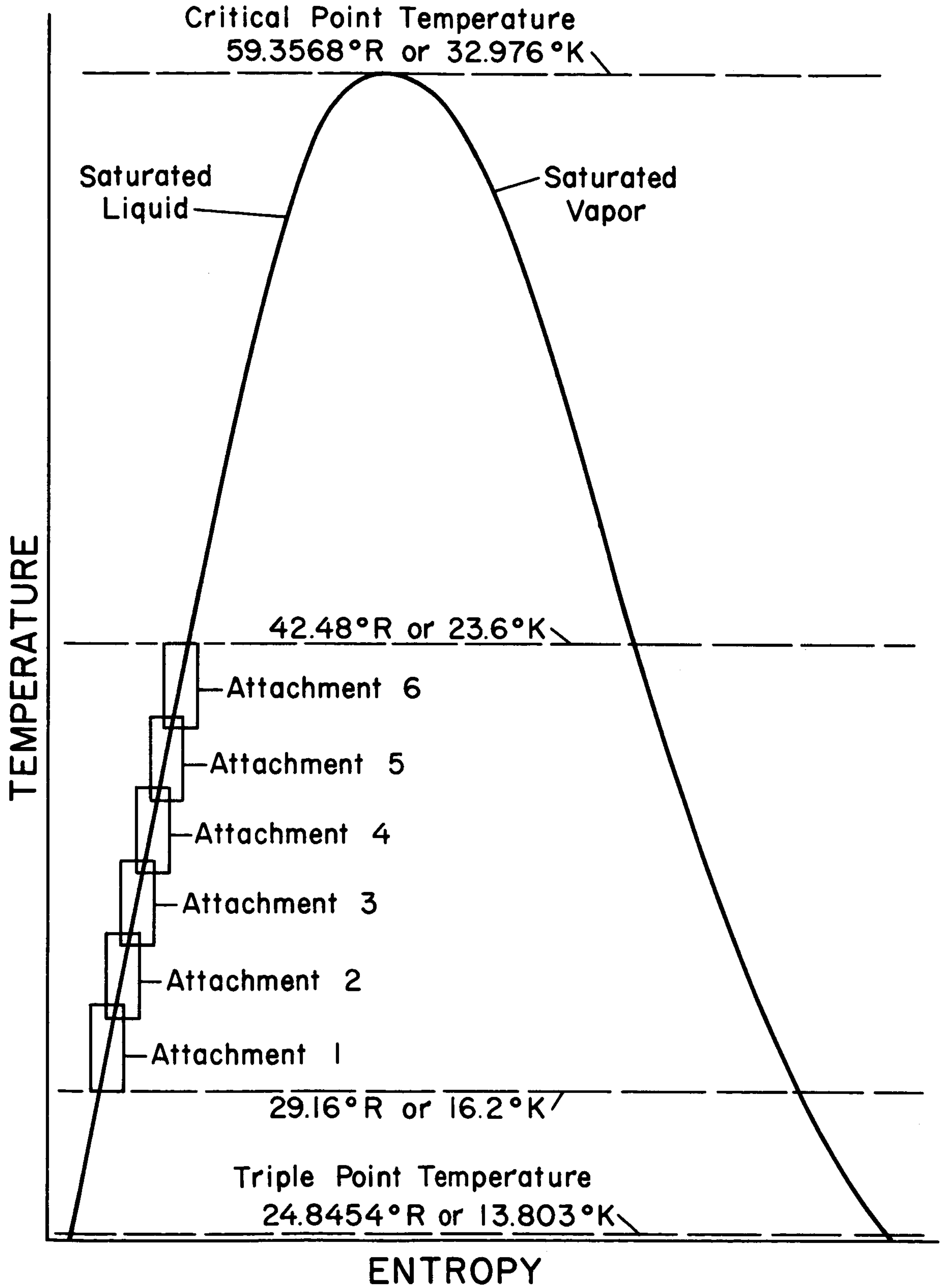


Figure 1. Range and Relative Location of TS Charts

## 2. Cryogenic Metrology (Instrumentation)

2.0 General Comments

Personnel contributing to the activities during this period were S. B. Lang, J. W. Dean, and R. J. Richards.

2.1 Temperature

2.1.1 Experimental Pyroelectric Studies

The experimentation has been completed and the progress report written. The final report on this work is in press.

2.2 Pressure

2.2.1 Piezoresistive Effects in Materials

The experimentation of this work is completed and the final report has been written. This final report is in press.

## 3. Consultation and Advisory Services

3.0 General Comments

Consultation and advisory services in the general field of cryogenic engineering have continued in several NASA program areas: Centaur (funded separately), Rover, and NERVA.

3.1 Centaur Program - Robert Arnett

Contact with NASA-Lewis Research Center personnel has been frequent. One trip to Cleveland-LeRC was made during this period.

3.1.1 Stratification and Pressurization

Manual check out of the various subroutines has been completed and the subroutines combined into a complete computer program. Calculations are in progress covering a range of ullage volumes and heat fluxes. A report covering the analysis, computer program, and typical results is in process and will be available in February 1967 as NBS Report 9266, "Mathematical Analysis of Thermal Stratification and Self Pressurization in a Closed Container." Copies will be available upon request.

3.1.2 Flight Data Analysis

Flight Data tapes of the AC-9 launch from the Tel-II and Pretoria stations have been received and reduced to graphical form. Complete analysis of the data is delayed pending the receipt of data types from other down range stations.

3.1.3 Helium Facility Study

Preliminary plans have been formulated to inspect and evaluate the helium recovery system at pads 36A and 36B, Cape Kennedy. A trip to the Cape to initiate this work is anticipated in the near future.

3.2 Rover Program - Alan F. Schmidt

Information pertaining to the selection and use of epoxy-based cryogenic adhesives and hydrocarbon analysis of a hydrogen gas stream was submitted to the CMF-9 group at Los Alamos.

3.3 NERVA Program - Alan F. Schmidt, Daniel H. Weitzel

A meeting with personnel from SNPO-C, General Dynamics, Aerojet-General, and Westinghouse Astronuclear was attended in Fort Worth, Texas on October 6 for the purpose of discussing thermal conductivity test project plans, test specifications, and experiment error analysis; tentative work schedules were reviewed at this time. Subsequently, modifications made in other areas of the radiation effects program have permitted an increased thermal conductivity effort for the next eight months. As a result, a meeting was called in Cleveland on December 19 to lay ground work for the revised program and for another conference in January, 1967; Weitzel attended this Cleveland meeting.

On October 24-26, NASA Contract R-45 program planning and project reviews were presented to Dr. Landon Nichols, the NASA (SNPO) technical monitor. In the course of discussing a new task concerning the determination of explosive characteristics of liquid hydrogen containing air as a contaminant, interest was renewed once again in a related topic of explosions involving irradiated cryogens. A general accumulation of information has been achieved in this area with the problem being signalled for continuing attention in the testing and use of nuclear reactors.

## 4. Cryogenic Flow Processes

### 4.0 General Comments

Personnel contributing to the project during the present reporting period were W. G. Steward, E. G. Brentari, and J. A. Brennan.

### 4.1 Analysis of Transfer Line Cooldown

An example of a computed pressure history for the first 1.4 seconds is shown in figure 2 along with a corresponding experimental curve. The calculated peak of 130 psia is for an inlet temperature, $T_{in}$, of 20.5°K; however, with all other variables fixed, $T_{in}$ of 19.6°K produced 180 psia, and $T_{in}$ of 21°K produced 100 psia peak pressures. Thus, a variation in the specified inlet liquid temperature of only 1.4°K produced an 80 psi change in computed peak pressure. This unexpected strong dependence on $T_{in}$ near the atmospheric boiling point is a possible explanation of the large variation of experimental peak pressures at supposedly the same operating conditions, since an inlet temperature variation of one or two degrees could have been present but not detected by the present instrumentation. Therefore, a much more sensitive germanium resistance thermometer will be used in future experiments to test the effect of small inlet temperature changes. The sensitivity is not present at higher saturation temperatures.

The computation proceeds satisfactorily as long as the time increment is kept small in the finite difference scheme. One hundred forty time increments of 0.01 seconds each were needed to produce the 1.4 seconds of flow time shown in figure 2 . This required 20 minutes on the CDC 3600 computer. To continue such meticulous computation for a 70-second cooldown would be quite impractical; therefore, the major effort at present is to speed up the computation of the final stages of cooldown.

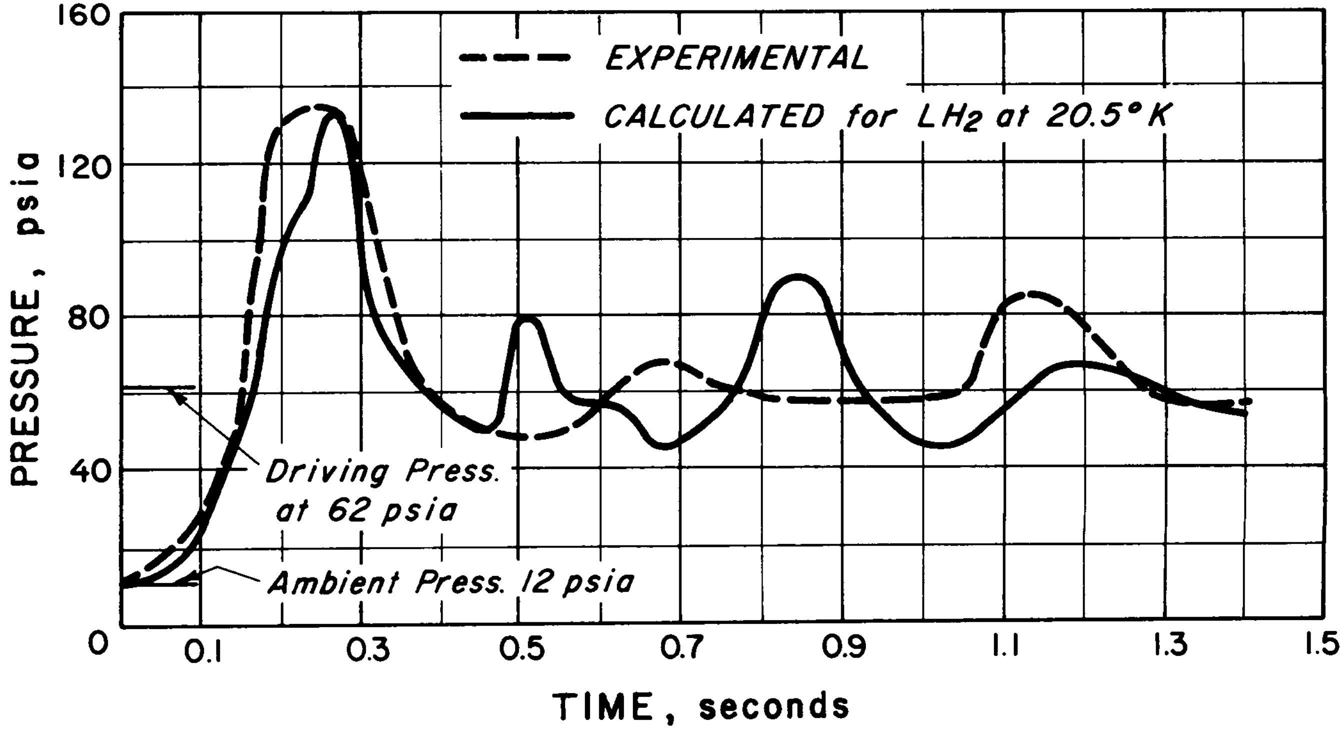


Figure 2. Pressure 20 ft. from the inlet of a 200 ft. pipeline

A paper entitled "Cooldown Time for Simple Cryogenic Pipelines" has been prepared. This paper presents an easy method of estimating cooldown time for long, well insulated pipelines by use of the graph of figure 3. Some of the calculated and experimental cooldown times are compared in figures 4, 5, and 6. The authors intend to submit the paper for the 10th Midwestern Mechanics Conference, August 1967. In figure 3:

$t$ = cooldown time

$u_s$ = speed of sound in the ambient temperature gas

$L$ = total length of pipe

$D$ = inside diameter of pipe

$\bar{f}$ = Blasius friction factor for the discharge gas, averaged over the process

$$\beta = 1 + \frac{\Delta h_w \rho_w A_W - \Delta h_\ell \rho_\ell A_f}{\rho_g A_f (\Delta h_\ell + \Delta h_{tot})}$$

$\Delta h_w$ = enthalpy drop of the pipe material

$\rho_w$ = density of the pipe material

$A_w$ = pipe solid cross-sectional area

$\Delta h_\ell$ = liquid enthalpy rise from the liquid inlet temperature to saturation at the inlet pressure

$\rho_\ell$ = liquid density

$A_f$ = flow cross-sectional area

$\rho_g$ = gas density at ambient temperature, inlet pressure

$\Delta h_{tot}$ = total fluid enthalpy rise from saturated liquid to ambient gas

4.2 Experimental

Experimental results obtained from tests with the 3/4-in. O.D. by 200-ft long transfer line were summarized and reported in NBS Report 9264 entitled "An Experimental Report on Cooldown of Cryogenic

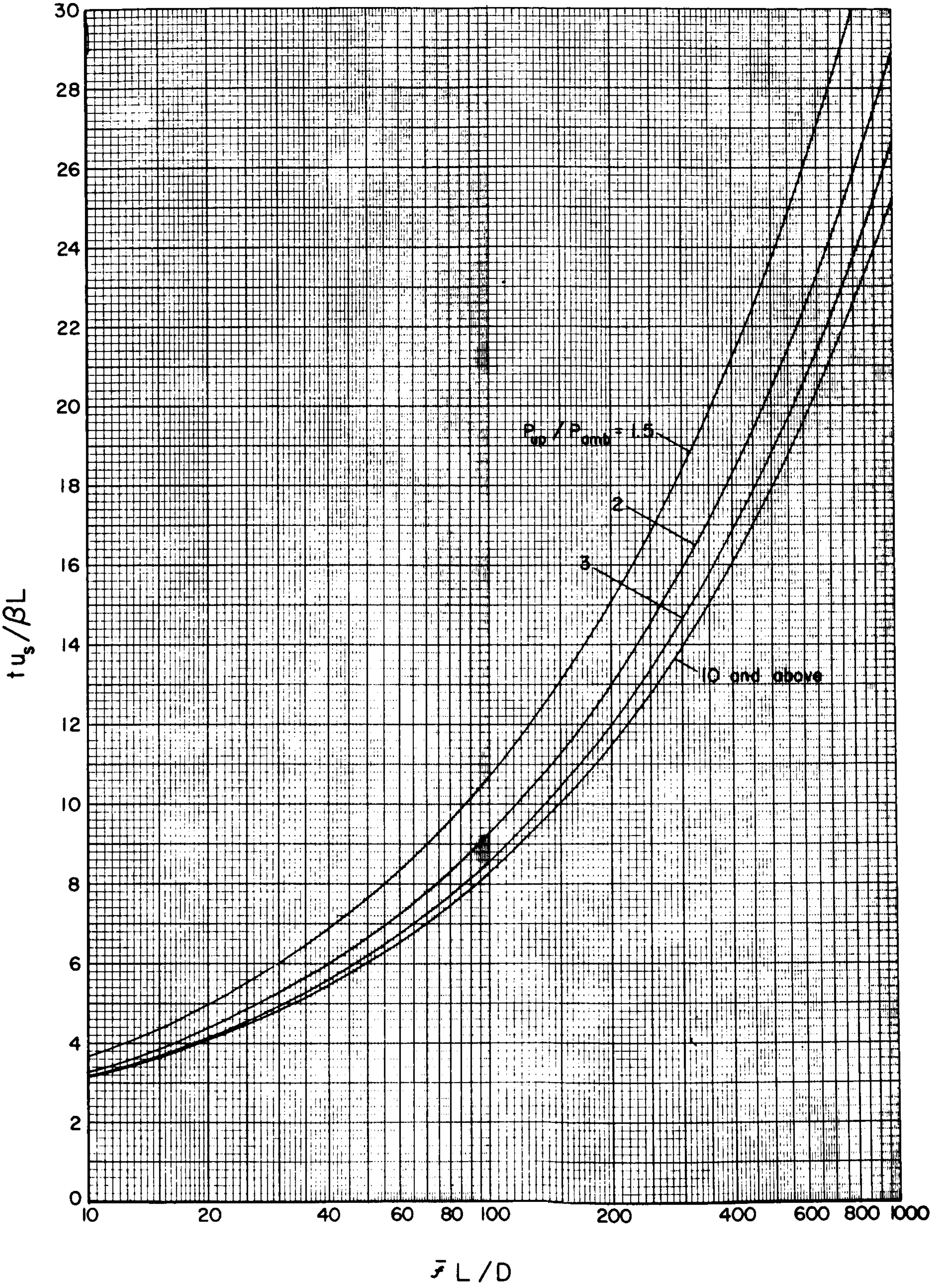


Fig. 3 Cooldown time parameters.

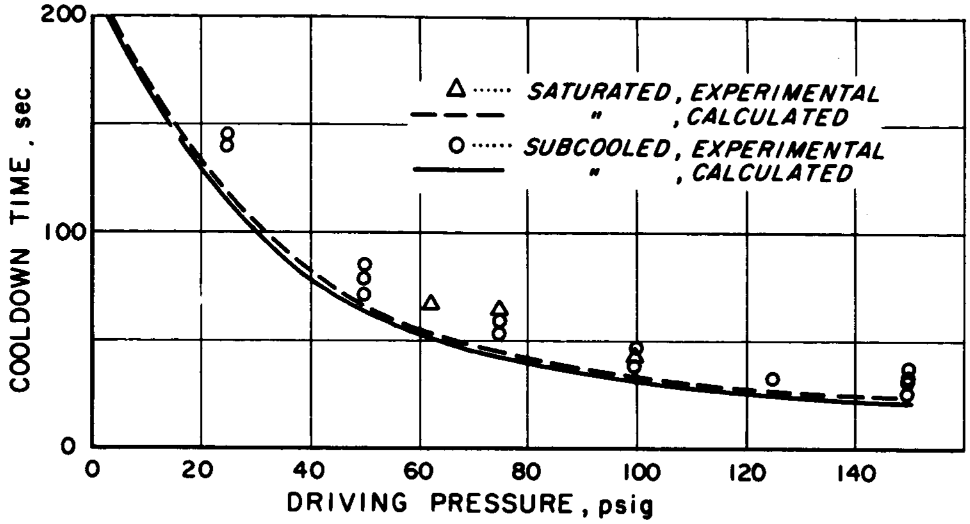


Fig. 4 Subcooled and saturated liquid hydrogen cooldown time--200 ft. pipeline.

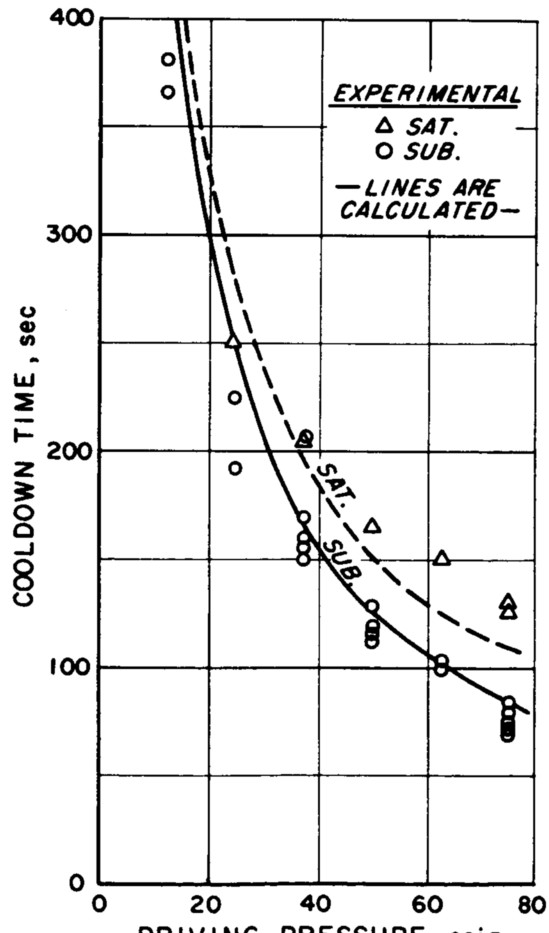


Fig. 5 Subcooled and saturated liquid nitrogen cooldown time--200 ft. pipeline.

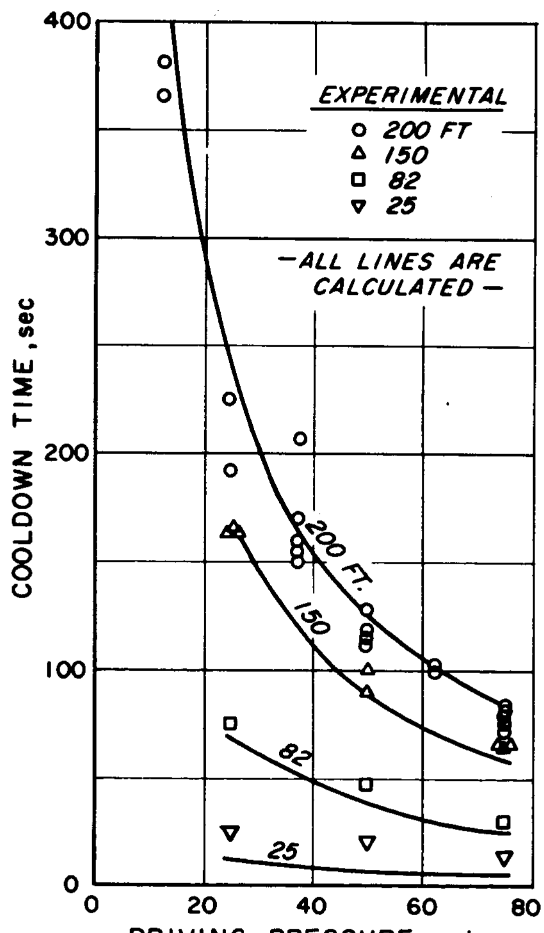


Fig. 6 Effect of pipe length on subcooled liquid nitrogen cooldown time.

Transfer Lines" by Brennan, Brentari, Smith, and Steward. This report is available and will be furnished upon request.

Results of the subcooled liquid nitrogen tests with shorter line lengths with and without end restrictions referred to in the last report are shown in figures 7 through 12. In figures 7 to 10 peak line pressure is shown as a function of transfer line length with driving pressure and end restriction as parameters. Figures 11 and 12 show cooldown time as a function of driving pressure with line length and end restriction as parameters.

The scatter in the peak line pressure was comparable to that obtained in earlier experiments with the 200-ft line. Scatter in the data plus the fact that only two or three runs were made at each condition makes any generalization risky but the data should be useful for comparisons with analytical predictions.

Cooldown times were more reproducible, but at low driving pressures the actual time of cooldown was difficult to define. Both line temperature and inlet flow rate were analyzed and the best estimate of actual cooldown is shown in the figures.

The 3/4-in. O.D. line has been removed and the 1/4-in. O.D. stainless steel line is being installed. In addition, some major changes are being made in the instrumentation in an effort to improve the accuracy and reproducibility of the data. It is hoped that some testing on the new line can be started during the next reporting period.

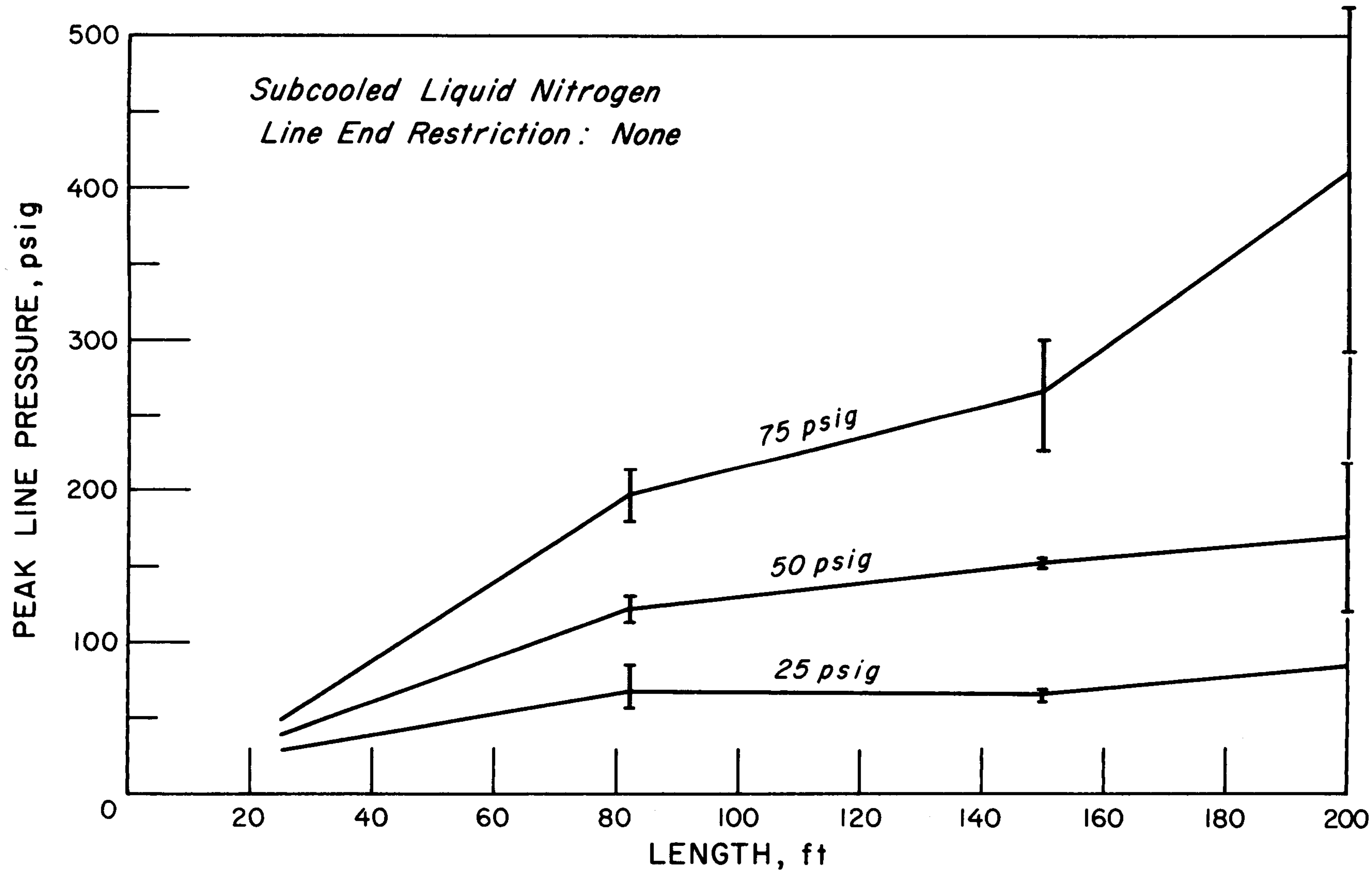


Figure 7. The effect of transfer line length on peak line pressure at various driving pressures. Subcooled liquid nitrogen.

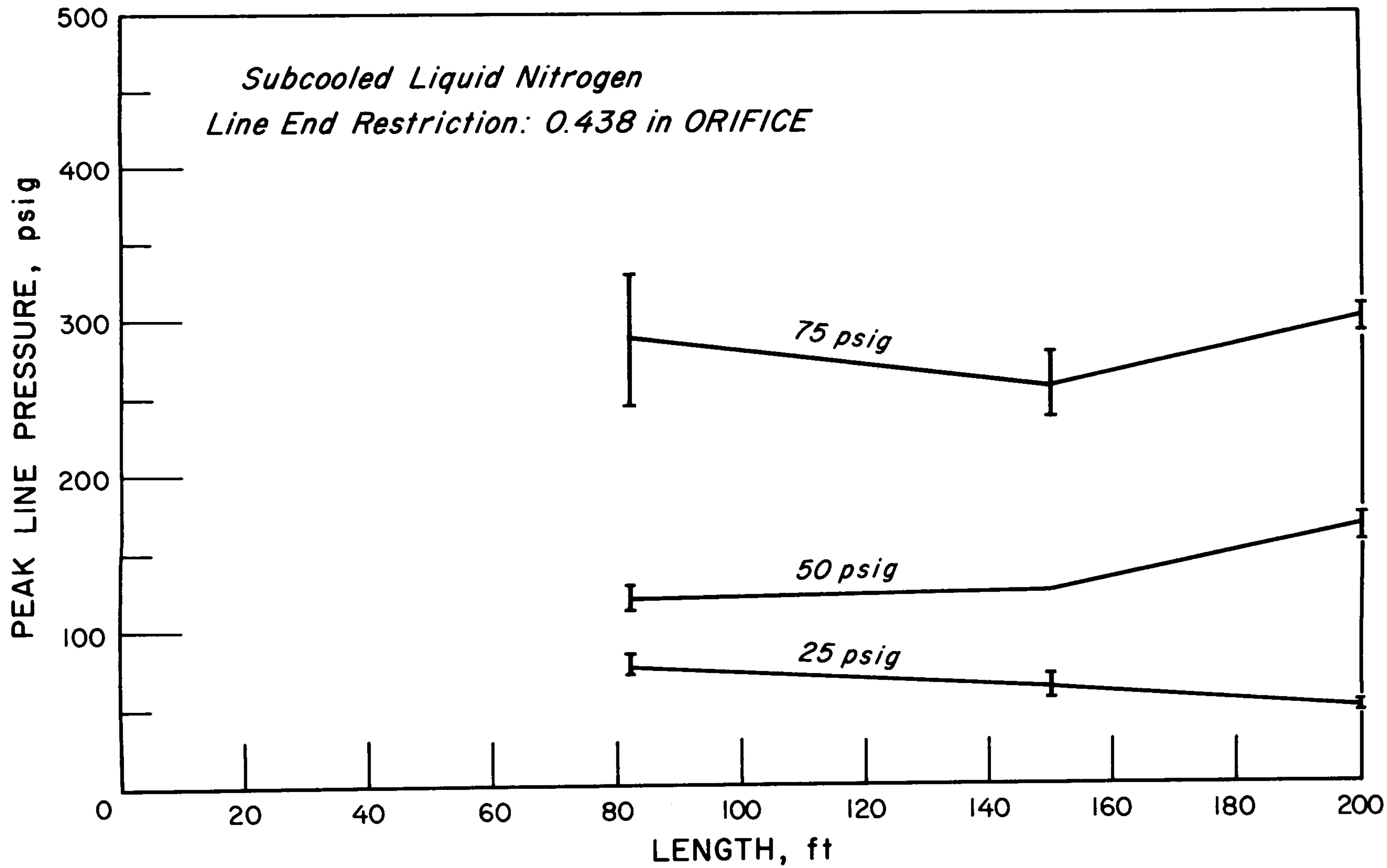


Figure 8. The effect of transfer line length on peak line pressure at various driving pressures with an 0.438-in end restriction. Subcooled liquid nitrogen.

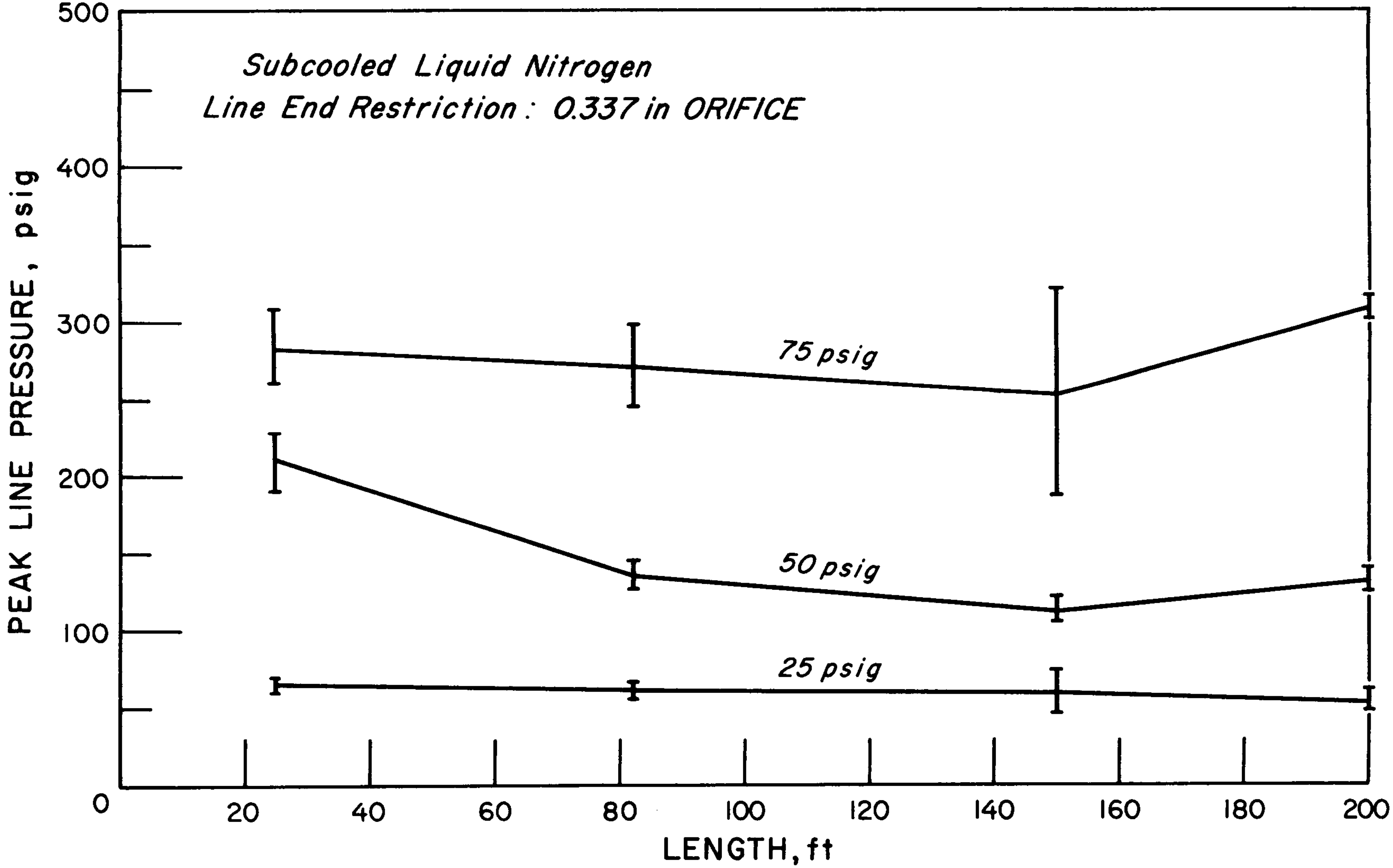


Figure 9. The effect of transfer line length on peak line pressure at various driving pressures with an 0.337-in. end restriction. Subcooled liquid nitrogen.

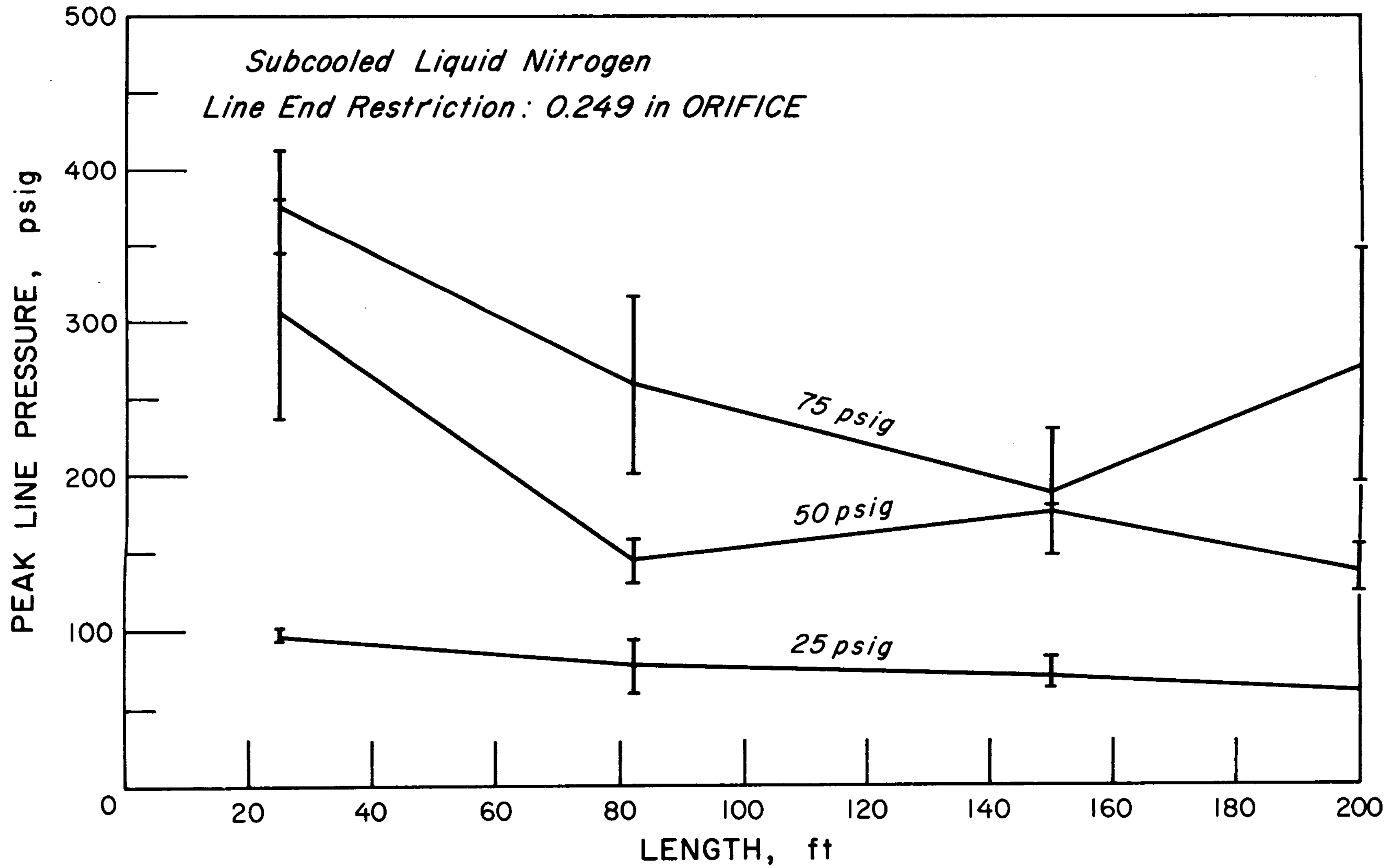


Figure 10. The effect of transfer line length on peak line pressure at various driving pressures with an 0.249-in. end restriction. Subcooled liquid nitrogen.

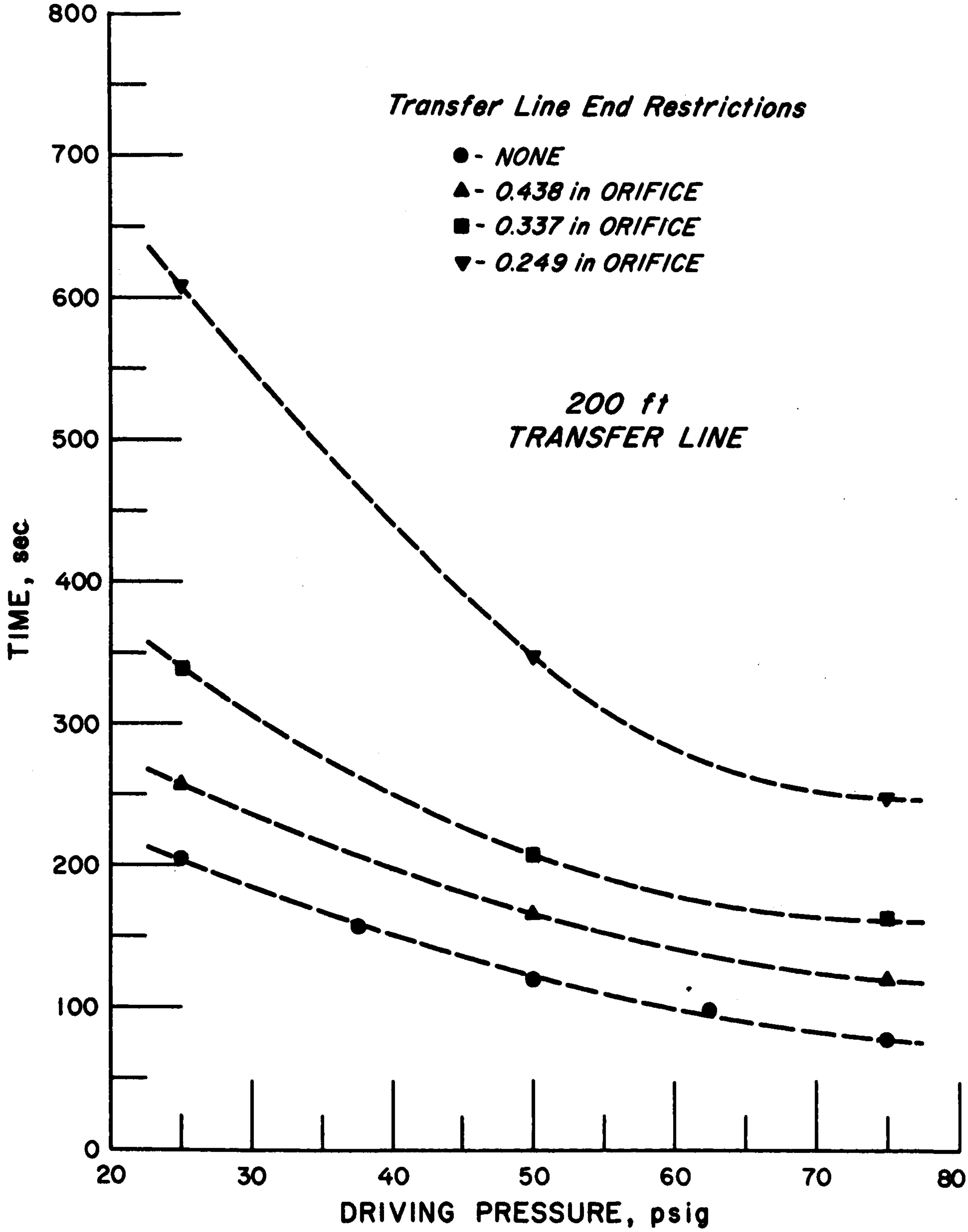


Figure 11. Cooldown time as a function of driving pressures for various line lengths and end restrictions. Subcooled liquid nitrogen.

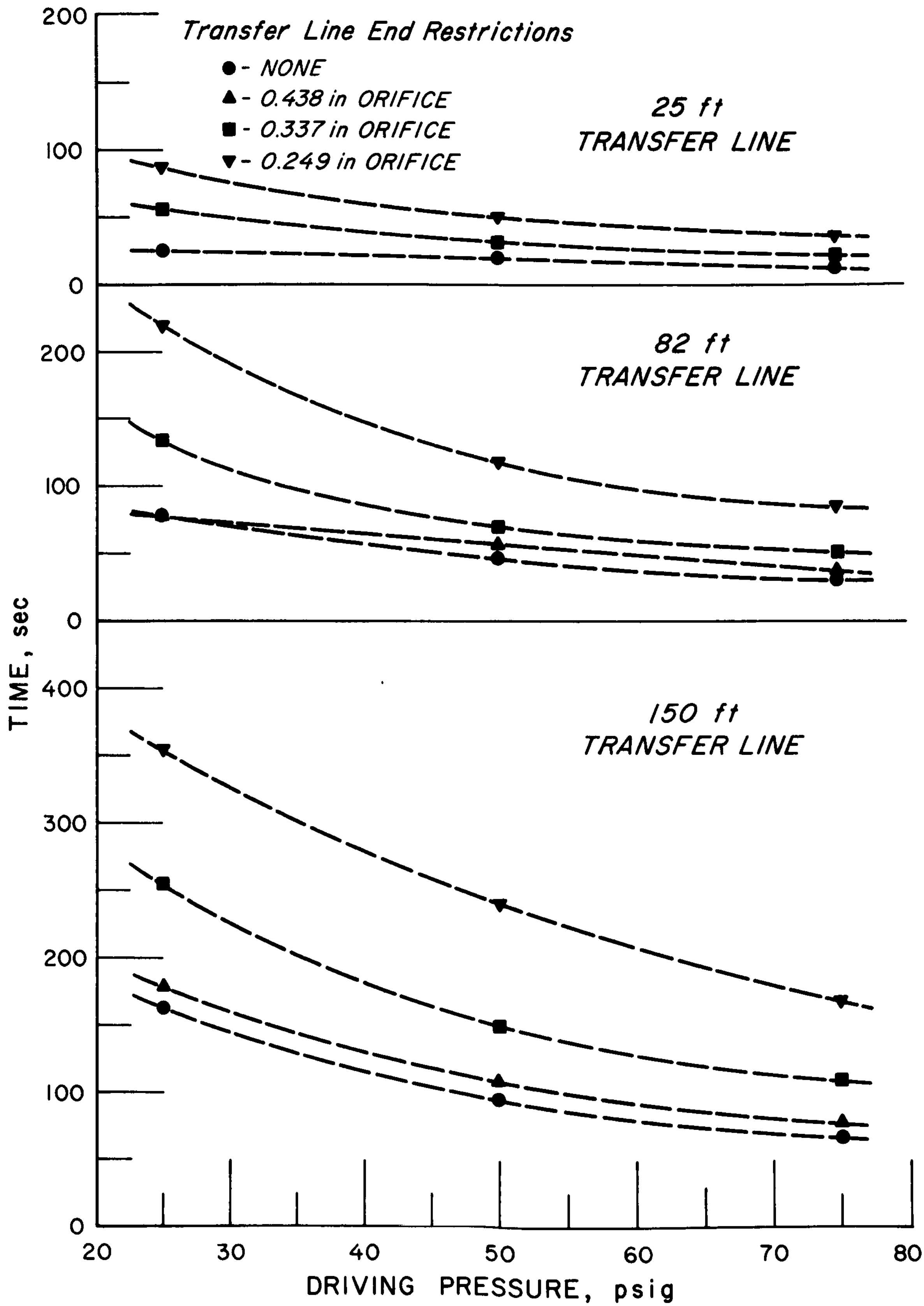


Figure 12. Cooldown time as a function of driving pressures for various line lengths and end restrictions. Subcooled liquid nitrogen.

## 5. Cryogenic Propellant Venting under Low Pressure Conditions

### 5.0 General Comments

Personnel contributing during this period were M. C. Jones and P. J. Giarratano.

### 5.1 Project Objectives

The goals of this project are three-fold:

(1) To measure heat transfer coefficients of solid-vapor mixtures of hydrogen or nitrogen flowing in a long straight tube below the triple point.

(2) To determine the effect of variable quality at the inlet of the tube.

(3) To determine the lower limit of heat flux required to maintain free-flow of the solid-vapor cryogenic fluid.

An analytical study to interpret the above is also underway.

### 5.2 Accomplishments During Current Reporting Period and Status of Project

NBS Report 9262 presents preliminary results of heat transfer measurements using nitrogen as the test fluid. At this time all experimental measurements have been completed for nitrogen and the test apparatus is being prepared for similar measurements using hydrogen as the cryogenic fluid.

# 6. Cryogenic Properties of Solids

## 6.1 Thermal Conductivity

### 6.1.1 General Comments

The objectives of this project are to determine the thermal conductivities of several aerospace alloys and standard reference materials from liquid helium temperature to above 120°K. The first material to be measured will be Titanium-5Aℓ-2.5 Sn. Other commercial alloys to be considered are Inconel-718, Steel A-286, Inconel-X and other alloys of interest to the NERVA II program.

The basic measurement method used by R. L. Powell, et al. [1] in previous work at this laboratory will be maintained. This is an absolute method consisting of the measurement of temperature at eight locations spaced one inch apart along a cylindrical sample. The heat flow is along the axis of the cylinder and is determined from the electrical power dissipated in a resistance heater. The sample will be vacuum insulated and radiation shielded. Also, considerable attention has been given to the elimination of heat loss along the leads connected to the sample.

Personnel contributing during this period were J. G. Hust, D. H. Weitzel, R. L. Powell, and N. C. Winchester.

### 6.1.2 Program Status

The mechanical system consisting of cryostat, pumps, and transfer line has been assembled and cold tested. The instrument panel rack is essentially complete with wiring from the terminal box to the instruments and switches and has been checked

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[1] Powell, R. L., W. M. Rogers, and D. O. Coffin, An Apparatus for Measurement of Thermal Conductivity of Solids at Low Temperatures, J. Res. NBS 59, 349-55 (1957), Research Paper 2805.

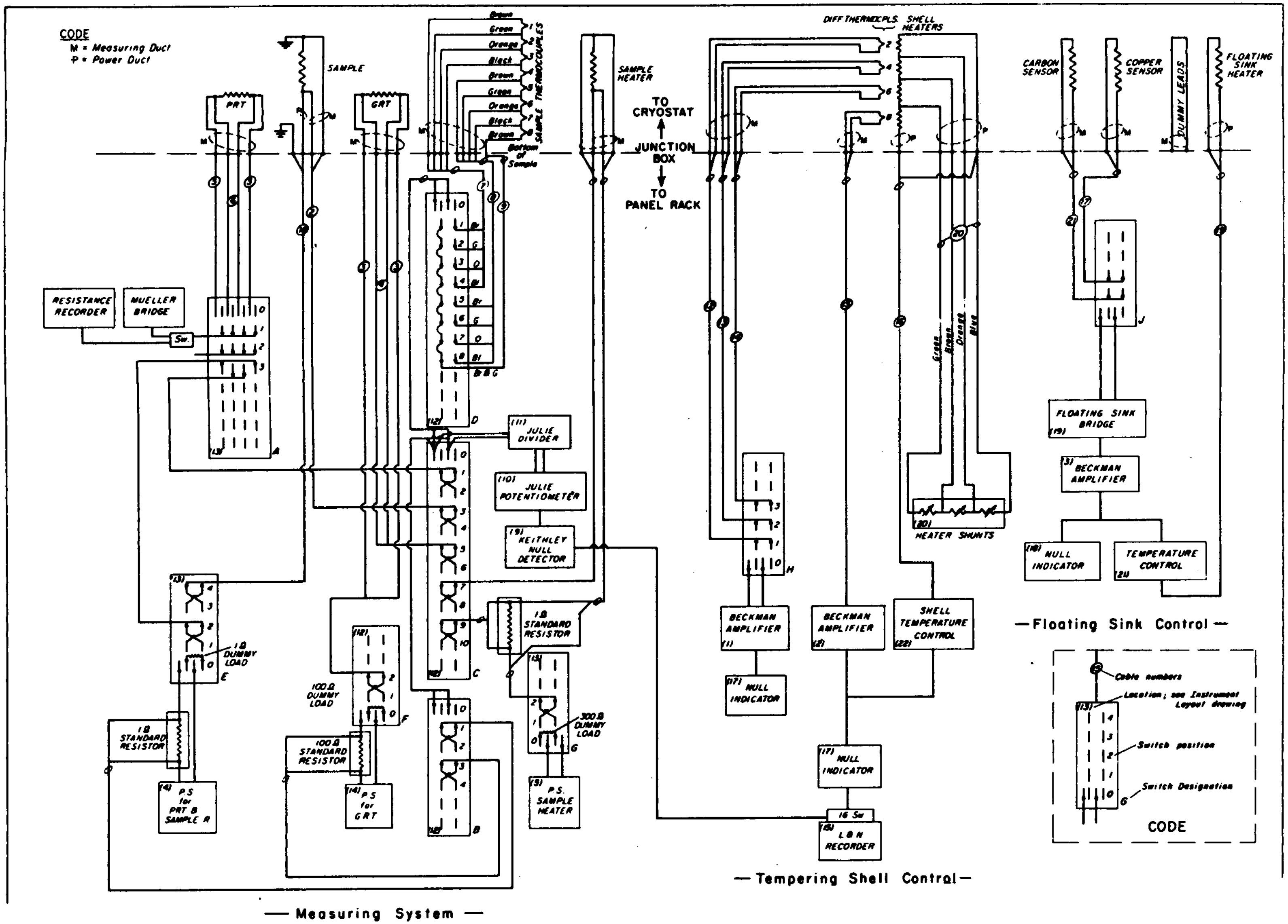


Figure 13. Thermal conductivity apparatus wiring diagram.

for continuity. A detailed wiring diagram is given in figure 13. The sample holder has been fabricated, wired and checked. With the completion of the sample holder wiring, D. H. Weitzel has transferred to another project and J. G. Hust has assumed full time duties on this project. The sample holder is illustrated in figure 14.

The Titanium alloy sample has been fabricated and the spacing and dimensions of the thermocouple holder groves have been carefully determined.

Measurements can begin after the following work is done. The sample holder is being wired into the system and is about 60% complete. The thermocouple holders must be mounted on the sample and the sample mounted into the sample holder. The entire system wiring will be rechecked before the vacuum system is closed for cold checking. The apparatus then will be tested both at room temperature and low temperatures.

The temperature regulator for the oil bath containing the standard resistors has been checked and is considered to be marginal if not inadequate. It has been decided to replace this regulator with a solid state switch unit similar to that being used by L. L. Sparks in the thermocouple calibration apparatus. This regulator also has been monitored for several hours and it regulates the temperature to within 0.01°C.

During the next reporting period the above steps will be completed and it is anticipated that measurements on Ti-5Aℓ-2.5 Sn at liquid nitrogen temperatures will be started.

## 6.2 Thermocouple Thermometry

### 6.2.1 General Comments

The immediate goal of the thermometry project is to furnish precision thermocouple calibrations for the thermal conductivity and thermal expansion work now in progress. Both groups are using

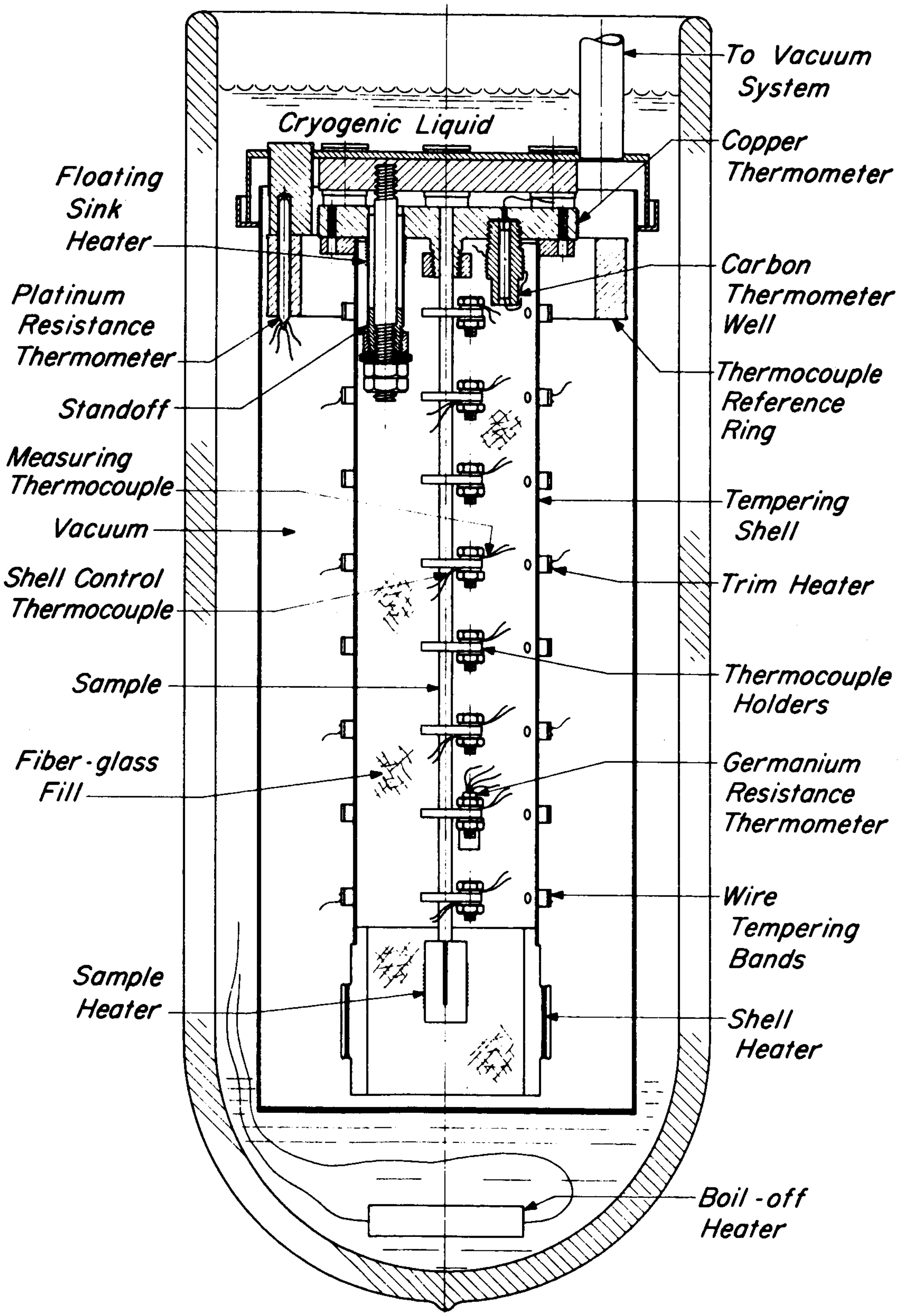


Figure 14. Thermal conductivity cryostat.

the relatively new and uncalibrated gold-iron thermocouple wire. Calibration of all commonly used low temperature thermocouple materials is continuing. The results will be used to establish calibration tables and eventually result in national standards for low temperature thermocouples. A versatile calibration system has been designed and constructed to allow calibration of other types of thermometers in the future.

Personnel contributing during this period were L. L. Sparks and R. L. Powell.

6.2.2 Program Status

The instrumentation for the thermocouple calibration apparatus is complete with the exception of the heater controller and the germanium resistance thermometer current supply. These units are being supplied by the NBS Cryogenic Metrology Section and will be delivered in January 1967. Upon receiving these units the entire data acquisition system will be tested. All major sub-systems such as the platinum resistance thermometer measure and control circuit, the thermocouple switching circuit, etc. have been tested as individual systems.

The cryostat wiring has been completed. The wiring involved the installation of twenty-two thermocouples, four platinum thermometers, and three germanium thermometers. The twenty-two thermocouple wires include the following materials: 1) copper, 2) Chromel, 3) Alumel, 4) constantan, 5) platinum, 6) "normal" silver, 7) silver 28 at.% gold, 8) gold 0.02 at.% iron, 9) gold 0.03 at.% iron, and 10) gold 0.07 at% iron. Although the wiring was simple conceptually, it was very difficult to accomplish due to the strain free mounting and thermal anchoring requirements of the thermocouple wires.

The calibrations for the reference thermometers have been carefully checked and adjusted. We have also furnished the reference thermometer calibration adjustment for the thermal conductivity of solids project.

### 6.2.3 Summary

The cryostat wiring and associated instrumentation is essentially complete. The "warm" check out of the entire system should be completed early in January. The next step will be a series of system checks at $LN_2$ temperature. Providing the $LN_2$ tests show no malfunction preliminary data can be taken by the middle of January.

It appears that the information required by the thermal conductivity program will be available when needed.

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